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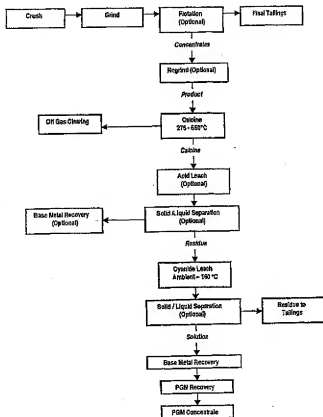
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(54) Title: PROCESS FOR EXTRACTING PLATINUM GROUP METALS



(57) Abstract: PGMs can be extracted from a source material by heat-treating the source material to form a residue containing PGMs in a cyanide leachable condition and, thereafter, cyanide leaching the residue using a solution containing cyanide to form a pregnant cyanide leach liquor containing PGMs in solution.

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Process for Extracting Platinum Group Metals

Field of the Invention

- The present invention relates to a process for extracting platinum group metals (PGMs) from a source material containing PGMs using heat treatment and subsequent leaching with a solution containing cyanide. In this specification, the expression "PGMs" is used to describe metals selected from the group comprising platinum, palladium, rhodium, ruthenium, osmium, iridium and mixtures thereof.
- The source material may contain base metals and the present invention also relates to a process for extracting base metals from the source material. In this specification, the expression "base metal" is used to describe metals selected from the group comprising copper, nickel, lead, tin, zinc, cobalt and mixtures thereof.

Background of the Invention

- PGMs may occur as discrete minerals or as dilute solid solutions typically in major sulphide minerals (for example, pentlandite, chalcopyrite or pyrrhotite). The separation chemistry of PGMs is amongst the most complex known with treatment being generally more complex as the sulphide or chromitite content of the ore increases. Often, gold is present in minerals rich in PGMs.

- Low sulphide PGM ores which contain small amounts of base metal sulphides are typically treated by fine grinding and bulk flotation to give a relatively low-grade PGM concentrate. The flotation reagents used are similar to those typically used for copper and nickel sulphides. The flotation concentrate is then dried before smelting to give a nickel-copper-iron-PGM matte. Smelting is a process by which a metal is separated from its ore in the presence of a reducing agent and a fluxing agent.

- The platinum group metals have a greater affinity with sulphide melts than with silicate melts and therefore partition with the matte phase rather than with the slag. The matte is "converted" while molten by blowing air into the matte to oxidise the matte and remove iron and some sulphur. The converter matte is then granulated or allowed to cool slowly so that discrete crystalline phases of nickel sulphide, copper sulphide, and a platinum group metal-containing magnetic phase are formed. This matte is then sent to a base metal

refinery where base metals such as copper, nickel and cobalt are removed and recovered by magnetic separation followed by acid leaching, or by direct acid leaching, leaving a high grade PGM concentrate. The high grade PGM concentrate is then sent to a PGM refinery which produces the individual PGM elements in metallic form. This route is expensive and

5 not altogether satisfactory for lower grade sulphide ores.

Medium sulphide ores which contain economic amounts of nickel plus copper (base metals) are typically treated by fine grinding and selective flotation, to give a nickel copper PGM sulphide concentrate. This concentrate is smelted in flash furnaces to give PGM-

10 containing mattes. The mattes are treated in various ways to give nickel and copper metal products plus PGM containing by-products which are sent to a refinery.

High sulphide ores which contain economic amounts of nickel and copper are also typically first treated by fine grinding and selective flotation, with or without magnetic separation, to

15 give separate nickel copper PGM and copper PGM sulphide concentrates. The nickel copper PGM concentrate which is usually low grade is calcined to remove some sulphur and then smelted in reverberatory or flash furnaces as for concentrates from medium sulphide ores.

Such prior art processes may also include gravity concentration in place of or in conjunction with the flotation step. A simplified block diagram of one current process flow sheet is provided in Figure 1 of the present specification. Recovery of PGMs by gravity

20 methods or by flotation may be difficult for ores with low sulphide mineral content concentration.

Conventional processes suffer from several limitations. Some PGM ores and in particular oxide ores from existing operations cannot be sufficiently upgraded by flotation to produce a concentrate which can be treated by a smelter. The same is often true for high chromitite

25 ores. Power consumption for the total process is high and the smelting process has difficulty in dealing with high chromitite ores, adversely effecting recoveries and costs.

30

PGM smelting capacity is concentrated in a limited number of countries, particularly South Africa, Canada, USA and Russia. Existing smelters are typically owned by a small number of companies which typically also operate mines associated with the smelters. Moreover,

transport of concentrates to the existing smelters is expensive, making projects remote from the existing smelters difficult to establish.

PGM refining capacity is less concentrated than the smelting capacity with numerous independent refineries operating in Europe and Asia in addition to those associated with the operating mines and smelters.

The market for total treatment of PGM concentrates is therefore less competitive than many other metals markets. Smaller projects cannot justify the large capital investment required for a smelter and refinery. There is therefore a need for an improved method for upgrading the PGM concentrates shipped to provide a high grade concentrate which would by-pass the smelter and be able to be shipped direct to a refinery. This would not only decrease the cost of production but increase the competitiveness of the market.

One alternative method to traditional processing that has been suggested in the prior art is selective leaching of PGMs from finely ground ore. There is no accepted solvent system for platinum group metals reported in prior art literature. Bromide, chloride, hydroxide, cyanide, bisulfide, thiosulphate, sulphite, and polysulphide ions and ammonia have all been suggested as suitable ligands for forming complexes with the platinum group metals. However, the stability and low solubility of some of these complexes and their reactivity with gangue minerals in the ore makes some of these ligands unsuitable as lixiviants for platinum group metals.

While PGMs are generally recovered from ores, there is also a significant market for recovery of PGMs from used automobile and other industrial catalysts and from computer and electronics scrap. There remains a need for an improved method of extracting PGMs from source materials other than ores.

It is to be clearly understood that, although prior art techniques are referred to herein, such reference does not constitute an admission that any of these techniques form part of the common general knowledge in the art in Australia or in any other country.

Throughout this specification, including the claims, the words "comprise", "comprises"

and "comprising" are used in a non-exclusive sense, except where the context requires otherwise due to express language or necessary implication, ie. in the sense of "including".

5 Summary of the Invention.

The present invention is based on the realisation that heat-treatment can be used to convert non-soluble PGMs present in a source material into a form which is soluble in a cyanide solution and that subsequent leaching in a solution containing cyanide can dissolve a substantial amount of the heat-treated PGMs.

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According to one aspect of the present invention, there is provided a process for extracting at least one PGM from a source material containing one or more PGMs, the process comprising the steps of:

15 heat-treating the source material to form a residue containing PGMs in a cyanide leachable condition; and

cyanide leaching the residue using a solution containing cyanide to form a pregnant cyanide leach liquor containing PGMs in solution.

20 Preferably, the step of heat-treating is conducted at a low temperature, for example below 550°C, to break down the material and liberate the PGMs from the material. Whilst it is possible for the heat-treating step to be conducted at high temperatures, for example above 550°C, it is preferred that the heat treatment is conducted below 550°C, more preferably below 500°C. At higher temperatures, the capital and operating expenditure is higher. Furthermore, it is more likely at higher temperatures, particularly in oxidising atmospheres, 25 for the surfaces of the PGMs to become passivated as they are liberated from the source material and thus render the PGMs less susceptible to cyanide leaching. Preferably, the step of heat-treating is conducted at a temperature in the range of 200°C to 550°C, more preferably 275°C to 500°C. Low temperature heat-treatment may be conducted in an oxidising or reducing atmosphere provided that the resultant residue contains PGMs in a cyanide leachable condition. Low temperature heat treatment in an oxidising atmosphere 30 has been found generally satisfactory. In this specification, the term "calcining" is used to describe the step of heat-treating in an oxidising atmosphere.

Optimum recoveries of PGMs have been found in test work when sulphide bearing minerals including PGMs have been calcined at a temperature in the range of 375°C to 425°C prior to cyanide leaching and hence calcination in this temperature range is particularly preferred. It is to be noted however that the particular heat treating conditions selected will be influenced by the precise nature of the source material.

Whilst the step of heat-treating may be conducted in an oxidising atmosphere or a reducing atmosphere at high temperature, it is preferred that high temperature heat treatment is conducted in a reducing atmosphere at a temperature between 550°C and 1000°C as a reducing atmosphere has been found to mitigate the problem of passivation of the surface PGMs at high temperature. Alternatively, the step of heat-treating may utilise a combination of oxidising and reducing conditions.

Preferably, the step of cyanide leaching is conducted at a temperature in the range of ambient and 160°C. It is preferred that the temperature does not exceed 80°C in order to minimise the breakdown of cyanide with increasing temperature. Thus, more preferably, the step of cyanide leaching is conducted at a temperature in the range of ambient to 80°C. PGMs can still be extracted using cyanide leaching at a temperature greater than 80°C, but doing so results in higher consumption of cyanide and thus higher operating costs. Alternatively, the cyanide leaching step may be conducted under pressure at a temperature within the range of 80°C and 160°C to increase the rate of metal dissolution and the overall recovery of metals.

The cyanide leaching process can take up to 120 hours or more depending on the type of source material. Preferably, the step of cyanide leaching is performed for 36-48 hours.

Preferably, a source of oxygen is injected during the cyanide leaching under pressure to improve the reaction kinetics.

The process may further comprise the step of repeating the step of cyanide leaching to increase the concentration of PGMs in the cyanide leach liquor.

The source material may also contain at least one base metal. When the source material

contains at least one base metal, the process preferably further comprises the step of acid leaching prior to the step of cyanide leaching to form a pregnant acid leach liquor containing at least one base metal in solution. Preferably, the step of acid leaching is conducted at a temperature between ambient and 200°C and a pressure between
5 atmospheric pressure and 20 bar. More preferably, the step of acid leaching is conducted at a temperature in the range of ambient and 100°C at atmospheric pressure.

Preferably, the step of acid leaching comprises the step of leaching in an acid selected from the group comprising sulphuric acid, hydrochloric acid, acid chloride or combinations
10 thereof. The particular acid selected will typically depend upon availability at a mine site with sulphuric acid being a common by-product of other metallurgical processes and thus often the most cost-effective acid available. The acid may be added directly as an acid or in the case of hydrochloric acid, the acid may be generated by the addition of sodium chloride, for example, and sulphuric acid to form the hydrochloric acid.

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It is to be noted that for a source material low in base metals, the step of acid leaching may not be required. For source materials containing high concentrations of base metal, the acid leaching step improves the recovery of the base metals and reduces cyanide consumption.

20 Recovery of base metals from the pregnant acid leach solution may be achieved using any number of conventional processes such as solvent extraction, ion exchange, electrowinning, reduction and precipitation or any combination thereof. Preferably, the process further comprises the step of recovering the at least one base metal from the pregnant acid leach liquor by solvent extraction, followed by electrowinning. An alternative preferred approach
25 is to recover the at least one base metal from the pregnant acid leach liquor by precipitation.

Preferably, the step of acid leaching is conducted at a pH within the range of 0.7 to 4.0. More preferably, the step of acid leaching is conducted at a pH within the range of 1 to 3. More preferably still, the step of acid leaching is conducted at a pH within the range of 1 to
30 1.5.

Preferably, said step of cyanide leaching is conducted at alkaline pH using a solution containing cyanide. More preferably, the step of cyanide leaching is conducted at a pH

within the range of 9 to 12, most preferably 9 to 10. It has been found that keeping the pH within the preferred range of 9 to 10 increases the recovery of PGMs, particularly platinum.

5 Preferably, the solution containing cyanide has a cyanide concentration less than 5%, more preferably less than 2%, and more preferably less than 1%. Typically the cyanide concentration will be within the range of 0.05% to 0.5% cyanide. Most preferably, the cyanide solution has a cyanide concentration in the range of 0.1% to 0.25% cyanide. Preferably, the solution containing cyanide contains sodium cyanide.

10 The solution containing cyanide may further comprise lime, caustic soda, peroxide, oxygen, lead nitrate or combinations thereof.

Preferably, the process further comprises the step of crushing and/or grinding the source material prior to the step of heat-treating. Where the source material is an ore, crushing
15 and/or grinding may be used to assist in liberating the PGMs from gangue. The term "gangue" is used in this specification to describe an unwanted substance which typically in a mineral would be one or more siliceous components. Gangue is desirably removed prior to heat-treating so as to reduce the quantity of material to be heat-treated and subjected to subsequent leaching operation(s) to both improve recovery and reduce operating costs.

20 Preferably, the step of crushing and/or grinding involves crushing and/or grinding the source material to a P80 in the range of 10 to 150 micrometres. The expression "P80" is used in this specification to refer to 80% of the material fed to a sieve of the nominated size passing through that sieve. More preferably, the step of crushing and/or grinding involves
25 crushing and/or grinding to a P80 in the range of 30 to 80 micrometres. More preferably still, the step of crushing and/or grinding involves crushing and/or grinding to a P80 in the range of 30 to 50 micrometres.

Where the source material contains gangue, the process preferably further comprises the
30 step of removing at least a portion of the gangue from the source material prior to the step of heat treating. The step of removing at least a portion of the gangue is preferably a flotation step which produces a flotation concentrate having a concentration of PGMs and/or base metals which is higher than the concentration before flotation.

The flotation step would be conducted under conditions conducive to the separation of the PGM minerals from the gangue. Reagents such as NaSH, copper sulphate, SIBX, SNPX, aeropromoters, sodium silicate and frothers might be added to assist in the flotation process.

- 5 The particular reagents such as collectors and suppressors, as well as other variables such as the pH selected for flotation, would depend on the type and grade of ore and the type of gangue minerals present in the ore.

- 10 It will be understood that any number of flotation cells arranged in series or parallel may be used, as indeed any other suitable apparatus or methods for separating ore from gangue, for example gravity concentration using jigging, shaking tables, or Knelson or Falcon concentrators, magnetic separation, optical sorting or electrostatic precipitation.

- 15 The flotation concentrate may be subjected to further grinding or milling followed by further stages of flotation and regrinding. The step of crushing and/or grinding the source material preferably occurs prior to the step of removing at least a portion of the gangue.

- 20 Preferably, the process further includes the step of grinding the flotation concentrate prior to the step of heat-treating. Preferably, the process further comprises the step of repeating the steps of removing and grinding to further improve the concentration of PGMs and/or base metals in the flotation concentrate prior to the step of heat-treating.

- 25 Preferably, the step of heat-treating is conducted in a fluidised bed or rotary kiln furnace. Although it is preferred that the step of heat-treating be conducted in a fluidised bed or rotary kiln furnace, it is to be understood that other types of heat treatment apparatus may be used depending on availability and provided the apparatus is capable of heat treating the source material to form a residue containing PGMs in a cyanide leachable condition.

- 30 Typically, the heat treating step will involve retaining the source material in a rotary kiln furnace under the selected heat treatment conditions for at least one hour. The preferred retention time during the step of heat-treating will be dependent upon a number of variables including the size and type of heat treatment apparatus, the size and type of the source material, and the selected heat treatment conditions.

Preferably, the process further comprises recovering PGMs from the pregnant cyanide leach liquor. The process may further comprise the step of removing solids from the pregnant cyanide leach liquor to form a cyanide leach filtrate. Any suitable means of solid/liquid
5 separation may be employed including filtration, counter current decantation, cyclone separation or a combination thereof.

The process may further comprise the step of recovering PGMs and/or base metals from the cyanide leach filtrate. The recovery step may comprise activated carbon adsorption,
10 solvent extraction, use of ion exchange resins, molecular recognition technology, electrowinning, reduction, precipitation, or a combination thereof.

Preferably, the process further comprises the step of recovering one or more base metals from the cyanide leach filtrate. Preferably, the step of recovering the base metals comprises
15 the step of solvent extraction.

PGMs may be recovered by the step of precipitating solids from the pregnant cyanide leach liquor to form a clarified cyanide leach liquor and separating the precipitates from the clarified leach liquor. The step of precipitating solids may comprise reducing the pH of the
20 pregnant cyanide leach liquor to precipitate PGMs (and base metals). Preferably, the pH is reduced to be within the range of 1 to 2.

Preferably, the process further comprises the step of recovering the cyanide from the pregnant cyanide leach liquor for re-use in the process. Cyanide may be recovered and
25 recycled to the process using conventional methods such as acidification/volatilisation/recovery (AVR); resin absorption from either slurry or solution; or solvent extraction. Using AVR, a slurry or solution is acidified and the hydrogen cyanide gas produced is removed by volatilisation in a stream of air. Gaseous hydrogen cyanide is then absorbed into an alkaline solution and recycled to the cyanide leaching
30 circuit. Alternatively, cyanide may be recovered by sulphide precipitation during the acidification stage. The precipitated metals are recovered from solution by solid-liquid separation and gaseous hydrogen cyanide is then volatilised from solution and absorbed into an alkaline solution.

Typically, the concentration of PGMs in the source material will be in the range of 1 gram to 1000 grams per tonne and the concentration of PGMs in the flotation concentrate will be in the range of 5 to 1000 grams per tonne.

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Preferably, the source material is a PGM ore, a sulphide mineral, a flotation concentrate or a spent catalyst.

Brief Description of the Drawings

- 10 In order to facilitate a better understanding of the nature of the invention, a preferred embodiment of the method for recovering platinum group metals will now be described in detail, by way of example only, with reference to the accompanying drawings, in which:

Figure 1 provides a flow chart showing a prior art method of recovering platinum group metals;

- 15 Figure 2 illustrates a flow chart of a preferred embodiment of the method in accordance with the present invention;

Figure 3 illustrates graphically the effect of calcine temperature on recovery of Pt+Pd+Au on the primary ore flotation concentrate of Example 1;

- 20 Figure 4 illustrates graphically the percentage recovery of Pt, Pd and Au over time for primary ore flotation concentrate calcined at 400°C with no regrind prior to cyanide leaching of Example 2;

Figure 5 illustrates graphically the percentage recovery of Pt, Pd and Au over time for primary ore flotation concentrate calcined at 400°C with a regrind to give a P80 of 33.5 µm of Example 2;

- 25 Figure 6 illustrates graphically recovery as a function of time with a regrind to give a P80 of 12.8 µm of Example 2;

Figure 7 illustrates graphically the effect of calcining temperature on the recovery of Pt, Pd and Au and the weighted average thereof for Example 3;

- 30 Figure 8 illustrates graphically the percentage recovery of Pt, Pd and Au over time for a sample calcined at 400°C for Example 3;

Figure 9 illustrates graphically the percentage recovery of Pt, Pd and Au over time for a whole of ore sample leached at 375 and 400°C;

Figure 10 illustrates graphically a typical flowsheet for a second preferred embodiment of the present invention;

Figure 11 illustrates graphically the effect of varying temperature on PGM recovery;

Figure 12 illustrates graphically the effect of cyanide concentration on PGM recovery;

5 Figure 13 illustrates graphically the effect of varying pH using lime on PGM recovery;

Figure 14 illustrates graphically the effect of varying pH using NaOH on PGM leach extraction;

10 Figure 15 illustrates graphically the effect of varying slurry dissolved oxygen levels on PGM recovery;

Figure 16 illustrates graphically the effect of varying $(\text{Pb}(\text{NO}_3)_2)$ addition on PGM recovery; and,

Figure 17 illustrates graphically the effect of pulp density on PGM recovery.

15 Detailed description of the Preferred embodiments

In the following illustrative examples the material treated is a sulphide ore. A typical flowsheet for treating such an ore is illustrated in Figure 2. Figure 2 shows a typical flowsheet for processing the ore according to a first embodiment of the present invention. The ore is subjected to crushing and grinding, followed by flotation, to separate a
20 concentrate rich in PGMs and base metals from the gangue which reports to the tailings. The concentrate may be reground and the ground product fed to a suitable heat treatment furnace such as a fluidised bed for calcining in the temperature range of 275°C to 550°C. The off-gas which may be rich in sulphur dioxide produced during the calcining process would typically be cleaned. The calcine residue may be subjected to an acid leach step if
25 the original ore is sufficiently rich in base metals to warrant an acid leaching step.

Following acid leaching, a solid/liquid separation process is conducted to remove an acid leach liquor rich in base metals. The base metals may then be recovered using any of the existing known processes. The solids removed during the solid-liquid separation stage
30 are then subjected to a cyanide leach at a range of temperatures between ambient and 160°C. Following cyanide leaching, a solid/liquid separation step is conducted again with the residue being sent to tailings and the pregnant filtered cyanide leach liquor being further treated to remove the base metals and/or the PGMs. Following the recovery

process, the PGM concentrate is then available for shipping to the end user.

In relation to Figure 10, a PGM concentrate or an ore containing PGMs is subjected to calcination and the off-gas from the calcination process may be treated using any known
5 process before being vented to atmosphere. Following calcination, the residue is repulped and reground and then subjected to a cyanide leach. After leaching, a solid/liquid separation stage is used to separate solids which are then repulped and sent to a residue storage facility and a clarified cyanide leach liquor to which is added acid to cause precipitation of the PGM's and base metals. During the acidification step the
10 cyanide volatilises and cyanide is recovered by adsorption in an alkali, allowing this to be recycled to the leaching stage.

A solid/liquid separation stage is used to remove the PGM's and base metals that have precipitated and any PGM's or base metals remaining in solution are recovered from the
15 solution by ion exchange prior to the solution being used as wash in the leach solid/liquid separation step or used for repulping the solid residue. The PGM/base metal precipitate is then subjected to an acid pressure leach to dissolve the base metals. This is followed by a solid/liquid separation to separate the PGM containing residue from the solution containing the base metals. The base metals are then recovered from the solution by the
20 addition of sulphide which causes precipitation of the base metals.

A solid/liquid separation stage is used to remove the base metals that have precipitated out and the clarified solution is then treated using cyanide volatilisation and cyanide
25 absorption by addition of an alkali to regenerate the cyanide for recycling to the leaching stage. Following cyanide volatilisation, the remaining liquor is subjected to an adsorption stage followed by elation and electro-winning to recover the PGMs. After the elation stage, a further sulphide may be added along with acid to cause precipitation of any remaining base metals.

30 Illustrative examples based on test work will now be presented to exemplify the present invention and should not be construed to limit the inventive method in any way. The test work is presented below in a series of tests which have been conducted either on oxidised ore, i.e. the ore that is closer to the surface and may have been oxidised, primary ore, which

is the below-surface ore nominally less than 60 metres, as well as whole ore. Throughout the test work the cyanide leach solution is a combination of sodium cyanide, lime, sulphuric acid and lead nitrate with the concentration in each example determined by the percentage of sodium cyanide in the cyanide leach solution.

5

Example 1: Primary Ore Flotation Concentrate Calcine - Leach

In the first series of tests, primary ore flotation concentrate with a nominal P80 feed size of 53 μm was calcined at a series of temperatures, namely 330°C, 400°C, 450°C and 500°C for two hours. The calcined ore was then subjected to a cyanide leach at 60°C for 48 hours at a pH of 9.5. The cyanide leach residue was reground to a P80 of 24 μm and subjected to a second cyanide leach under the same conditions. Figure 3 shows the effect of calcine temperature on the recovery expressed as the weighted average of Pt plus Pd plus Au. As can be seen clearly from Figure 3, the best results were obtained for calcining at 400°C with Pt recovery of 72.7%, Pd recovery of 91.8% and Au recovery of 99% after 48 hours.

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The total recovery of Pt, Pd, Au, Ni, Co and Cu are shown in Table 1 below.

Table 1

Element	330°C	400°C	450°C	500°C
Pt	39.0	72.7	60.9	14.0
Pd	89.5	91.8	92.1	82.2
Au	75.3	99.0	99.5	98.8
Ni	54.9	43.8	37.2	36.0
Cu	29.7	66.1	45.6	51.3
Co	20.4	20.5	12.3	12.0

20

Example 2

Primary ore flotation concentrate with a P80 size of 53 μm was calcined at a temperature of 400°C for two hours and the effect of a subsequent regrind prior to cyanide leaching was assessed. Tests were conducted without regrind, with a regrind P80 size of 33.5 μm and a third test with a P80 regrind size of 12.8 μm . Subsequent cyanide leaching was conducted at 60°C for up to 48 hours at a pH of 9.5 and the results are presented below in Table 2. Figure 4 illustrates the percentage metal extraction of Au, Pt and Pd as a function of time with no regrind. Figure 5 illustrates the percentage metal extraction of Au, Pt and Pd as a function of time with a regrind P80 of 33.5 μm . Figure 6 illustrates the percentage metal extraction of Au, Pt and Pd as a function of time with a regrind P80 of 12.8 μm .

Table 2

Element	No Regrind	Regrind P80 33.5 μm	Regrind P80 12.8 μm
Pt	64.1	84.3	81.4
Pd	86.6	92.9	95.3
Au	97.3	99.2	99.4
Ni	47.5	50.6	64.9
Cu	77.0	79.9	81.4
Co	22.8	32.8	49.7

These figures illustrate that the recovery can be improved with finer grinding prior to cyanide leaching.

Example 3: Oxidised Ore Flotation Concentrate Calcine- Leach

Tests were conducted on oxidised ore flotation concentrate subjected to calcining at a range of temperatures followed by cyanide leaching. The oxidised ore had a P80 feed size of 53 μm . Calcining was conducted at 350°C, 400°C and 450°C for two hours with a subsequent regrind to bring the P80 size to 20 μm . The samples were then subjected to a cyanide leach at 60°C for 48 hours at a pH of 9.5 and the recoveries are presented in Table 3 and Figure 7.

Table 3:

Element	350°C	400°C	450°C
Pt	45.4	64.4	46.2
Pd	85.1	83.5	71.4
Au	98.0	99.4	99.3
Ni	10.5	20.1	10.5
Cu	54.8	52.2	54.8
Co	10.9	15.5	10.9

Figure 8 illustrates the percentage recovery of Au, Pt, Pd and the weighted average of Pt + Pd + Au as a function of time for calcining at a temperature of 400°C.

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Example 4: Acid Leaching of Calcined Oxidised Ore Flotation Concentrate

Tests were conducted to assess the effect of a subsequent acid leach following calcining at 400°C. An oxidised ore flotation concentrate with a P80 size of 53 µm was subjected to calcining at 400°C for two hours. A regrind to give a P80 size of 20 µm was conducted on the sample that was not subjected to a subsequent acid leach, but no regrind was conducted on the sample to be acid leached. Acid leaching was conducted at a pH of 1.5 with sulphuric acid at ambient temperature for eight minutes. Both samples were then subjected to a cyanide leach at 60°C for 48 hours with a pH of 9.5. The results are presented in Table 4 below.

15

Table 4

Element	400°C calcine plus regrind to P80 20 µm	400°C roast, no regrind and acid leaching
Pt	54.3	47.2
Pd	85.0	87.4
Au	99.3	98.9
Ni	15.0	18.2
Cu	43.3	64.4
Co	11.4	44.5

The effect of acid leaching is to increase the recoveries of the base metals Ni, Co and Cu without unduly affecting the recovery of Pt and Au. Surprisingly, the Pd recovery has improved following subsequent acid leaching.

20

Example 5 - Oxide Ore Calcine Leach Tests for Whole Ore

Tests were conducted on oxide ore with a P80 feed size of 38 μm to assess the effect of calcining temperature being varied between 375°C and 400°C. Calcining was conducted for two hours with no subsequent regrind or acid leaching. Subsequent cyanide leaching was conducted at 60°C for 48 hours at a pH of 9.5 with the results presented in the following Table 5.

Table 5

Element	375°C	400°C
Pt	8.2	4.9
Pd	73.6	99.5
Au	98.5	66.4
Ni	31.6	7.1
Cu	35.5	35.0
Co	2.3	2.5

Figure 9 illustrates the percentage recovery as a function of time for the results presented above in Table 5.

Example 6 - Effect of Leach Temperature

The results of tests conducted to evaluate the effect of varying cyanide leach temperature are summarised in Table 6 below and plotted in Figure 11.

The results indicate that PGM metal recoveries increase up to a cyanide leach temperature of 60°C and plateau out, slightly decreasing up to 75°C. Base metal recovery varied slightly over the range tested but tended to decrease at higher temperature. 60°C has thus been selected as the preferred leach temperature.

Table 6

Leach Conditions		Extraction %						
Leach Temp °C	Leach Time, hours	Pt	Pd	Au	PGM	Cu	Ni	Co
50	48	48.8	84.0	96.3	70.4	62.4	21.5	12.4
60	48	81.1	89.0	94.4	86.1	59.2	23.2	17.0
75	48	81.1	88.1	95.8	85.7	57.8	22.1	18.1

Example 7 - Cyanide Leach Concentration

- 5 A series of leach tests were conducted on ground calcine, at pH 9.5, 60°C and dissolved oxygen levels of +13 ppm for 48 hours over a range of cyanide solution concentrations.

The results summarised in Table 7 and illustrated in Figure 12.

Table 7

Leach Conditions		Leach Extraction %						
Soln. NaCN	Leach Time, hours	Pt	Pd	Au	PGM	Cu	Ni	Co
0.2%	48	81.1	89.0	94.4	86.1	59.2	23.2	17.0
0.05%	48	64.4	76.5	84.4	72.2	6.5	2.6	2.8
0.1%	48	77.2	86.9	92.6	83.3	20.7	10.4	7.3
0.4%	48	79.0	90.3	94.8	86.3	67.3	32.4	24.0

10

From Table 7 and Figure 12, it is apparent that 0.2% NaCN concentration produced the highest Pt recovery and Pd and Au recoveries only increased marginally at 0.4% NaCN. Thus 0.2% NaCN concentration was selected as optimum. Base metal recoveries were slightly higher at the maximum cyanide strength tested. The extra cyanide costs at 0.4% NaCN were not justified by the small additional recoveries.

15

Example 8 - Slurry pH with Lime

The effect of pH on metal recoveries was evaluated using lime as pH modifier. The average pH recorded throughout the tests was used as basis of the evaluation.

- 20 The results are summarised in Table 8 and plotted in Figure 13.

Table 8

Leach Conditions		Leach Extraction %						
pH	Leach Time, hours	Pt	Pd	Au	PGM	Cu	Ni	Co
9.1	48	81.1	89.0	94.4	86.1	59.2	23.2	17.0
9.5	48	78.8	88.2	96.2	84.9	58.6	22.4	12.7
9.8	48	76.8	86.6	75.0	81.1	55.4	20.7	12.6

In the pH range tested the results indicate that pH of 9.1 is optimum for Pt and Pd and Au recovery is optimum at pH 9.5 but only marginally lower at pH 9.2. Base metal recoveries were greatest at the lowest pH tested.

Example 9 - Slurry pH with NaOH

The effect of pH, on metal recoveries was evaluated using caustic soda as pH modifier. The average pH recorded throughout the tests was used for comparison. The results are summarised in Table 9 and plotted in Figure 14.

Table 9

Leach Conditions			Leach Extraction %						
pH Modifier	pH	Leach Time, hours	Pt	Pd	Au	PGM	Cu	Ni	Co
Lime	9.1	48	81.1	89.0	94.4	86.1	59.2	23.2	17.0
5.6 kg/t NaOH	9.6	48	66.8	90.0	96.1	80.6	58.3	22.9	15.0
8.2 kg/t NaOH	10.5	48	73.0	90.7	88.7	83.1	53.7	20.6	14.8
10.8 kg/t NaOH	10.8	48	70.7	90.5	90.1	82.0	52.9	19.9	15.7

In the pH range tested the results indicate that Pt recovery is optimum at 10.5, Au at pH 9.6 and Pd at 10.5. The best Pt recovery with caustic soda, however, is 8% less than achieved with lime. The best Pd and Au recoveries achieved with caustic soda are 90.7% and 96.1% compared to 89.0% and 96.2% achieved with lime. Lime produced higher base metal recoveries than caustic soda.

Lime was thus determined to be the preferred pH modifier.

Example 10 - Level of Dissolved Oxygen in Leach Slurry

The concentration of dissolved oxygen (DO) in the leach slurry was varied by adjusting the feed rate of oxygen or air into the head space of the sealed leach tank. The effect of varying DO levels on metal recovery is summarised in Table 10 and the results plotted in

5 Figure 15.

Table 10 - Effect of Varying Slurry DO Level on PE Leach Extraction

Leach Conditions			Leach Extraction %						
Oxygenation	Average DO ppm	Leach Time, hours	Pt	Pd	Au	PGM	Cu	Ni	Co
Standard	13.4	48	81.1	89.0	94.4	86.1	59.2	23.2	17.0
DO at 5 ppm	5.9	48	81.4	88.5	81.8	84.8	58.1	23.4	15.5
DO at 10 ppm	9.9	48	77.5	86.9	93.8	83.4	56.3	21.9	15.6
Air atmosphere	2.8	48	75.4	88.3	90.8	83.1	57.2	22.4	12.6

The results indicate that Pt recovery was optimum and stable over the DO range 6 to 13 ppm, and Pd and Au recoveries were optimum in the range 10 to 13 ppm. Base metal

10 recoveries were similarly optimum over the DO range of 6 to 13 ppm.

A DO level of 10 ppm was selected as optimum overall.

Example 11 - Use of Lead Nitrate

The effect of lead nitrate on metal recovery is summarised in Table 11 and the results

15 plotted in Figure 16.

Table 11

Leach Conditions			Extraction %						
Notes	Average DO ppm	Leach Time, hours	Pt	Pd	Au	PGM	Cu	Ni	Co
50 g/t Pb(NO ₃) ₂	13.4	48	81.1	89.0	94.4	86.1	59.2	23.2	17.0
0 g/t Pb(NO ₃) ₂	15.8	48	76.7	88.6	95.5	84.1	58.8	23.0	14.1
50 g/t Pb(NO ₃) ₂	16.6	48	80.1	86.6	95.9	84.6	59.3	23.4	15.4
100 g/t Pb(NO ₃) ₂	14.6	48	77.8	88.1	95.8	84.3	59.2	23.2	13.7
200 g/t Pb(NO ₃) ₂	15.4	48	75.6	85.8	96.6	82.2	61.7	23.9	15.6

The results indicate that Pt and Pd recovery peaked in the 0 to 50 g/t lead nitrate addition rate range and Au recovery increased above this addition rate. The total PGM recovery is

within 0.5% over the 0 to 100 g/t lead nitrate addition rate and decreases at greater addition rates. No specific trends in base metal recoveries were observed with different lead nitrate addition rates. Given the operating costs of the lead nitrate and minimal indicated recovery gain the use of the reagent is not justified in this example.

5

Example 12 - Acid Leach Tests

- 10 The acid leach tests were conducted on concentrates after calcining in a Midrex rotary kiln at 400°C. The tests were done to see what effect the calcining would have on base metal recovery following leaching with sulphuric acid. The results as presented in Table 12 demonstrate reasonably low base metal recovery, particularly nickel.

Table 12

Feed Material		Calcine Conditions			Leach Conditions			Leach Extraction %		
Grind P ₈₀	Float ref	Calcine Temp °C	Furnace	Calcine time h	Leach Temp °C	Solution pH	Leach Time h	Cu	Ni	Co
-	Concentrate 1	400	Rotary	1	25	1.5	8	57.1	12.1	43.1
38 µm	Concentrate 2	400	Rotary	2	60	1.6	4	61.5	25.2	63.8

15

Example 13 - Acid Leaching

A series of tests were conducted as acid (H₂SO₄ and HCl) leaches on concentrates in order to evaluate the potential for base metal recovery prior to calcination and the effect of the acid leach on the downstream calcining and PGM leaching and recovery.

20

Tests were conducted on H₂SO₄ leaches at pH 1.5 and the tails dried and fed to calcining / cyanide leach tests. Base metal recoveries were generally poor with copper, nickel and cobalt recoveries in the ranges, 32 to 44%, 9 to 13% and 13%, respectively. The results are summarised in Table 13.

- 25 Tests also investigated hydrochloric acid leaches, following calcination in the presence of sodium chloride. The base metal extractions in the acid leach were very low, with copper, nickel and cobalt all yielding less than 10% recovery. The results are also summarised in Table 13.

Tests were also conducted to evaluate sulphuric acid leaching of calcines produced in the Midrex rotating kiln with 2 hours calcining times. The base metal extractions were disappointing, with the highest recoveries being 61.5% and 63.8% for copper and cobalt, respectively. The results of these tests are also summarised in Table 13.

5

Table 13

Notes	Leach Conditions				Leach Extraction %			Leach Reagents	
	Calcine Grind F ₈₀	Leach Temp °C	pH	Leach Time, hours	Cu	Ni	Co	H ₂ SO ₄ kg/t added	HCl kg/t added
Acid leach tails feed to test H3694	No Calcination	amb	1.5	4	32.7	13.1	13.2	258	
Tails not cyanide leached	No Calcination	45	1.5	4	No base metal assays			279	
Tails not cyanide leached	No Calcination	60	1.5	4	No base metal assays			345	
Acid leach tails feed to test H3696	No Calcination	60	1.5	4	44.7	9.2	12.7	152	
100 g NaCl to calcination feed	36 µm	60	1.5	48	0.11	5.41	5.8		336
50 g NaCl to calcination feed	36 µm	60	1.5	48	0.35	9.04	6.5		297
148.4 kg/t H ₂ SO ₄	-	25	1.5	8	57.1	12.1	43.1	148	
217.3 kg/t H ₂ SO ₄	38 µm	60	1.6	4	61.5	25.2	63.8	217	

Example 14 - Leach Slurry Density

- 10 A series of leach tests were conducted using standard conditions at different slurry densities. The results are summarised in Table 14 and plotted in Figure 17.

Table 14 - Effect of Pulp Density on PGM Leach Extraction

Slurry Density	Leach Conditions		Leach Extraction %						
	pH	Leach Time, hours	Pt	Pd	Au	PGM	Cu	Ni	Co
45% w/w	9.1	48	81.1	89.0	94.4	86.1	59.2	23.2	17.0
40% w/w	9.2	48	80.7	91.3	97.6	87.2	63.3	24.5	16.6
50% w/w	9.1	48	79.5	88.0	94.0	85.0	45.5	18.6	12.8

- 15 The results indicate an optimum Pt recovery at 45% solids and very minor decline in Pd and Au recoveries with increasing density. Base metal recoveries, particularly Cu, were generally best at the lowest pulp density. Evaluation of slurry density on leach tank costs, cyanide costs and down stream benefits from higher tenor solutions indicates that 50%

solids is the preferable slurry density to be used.

Now that preferred embodiments of the method of extracting PGMs in accordance with the present invention has been described in detail, it will be apparent that it provides a number

5 of significant advantages, including the following:

- a) the ability to treat oxide ores which could not be treated by the traditional process routes.
- b) the ability to treat high chromitite ores which could not be treated by the traditional process routes.
- 10 c) production of a PGM concentrate which can be sold direct to a refinery, providing a reduction in transport costs; higher payable metal; larger market for the product providing more competitive price; reduced time between shipping concentrate and receiving payment; reduced power consumption and lower total cost of production.
- 15 d) the ability to develop operations without the need to construct a smelter or incur significant expenses in shipping concentrates.

Numerous variations and modifications will suggest themselves to persons skilled in the metallurgical engineering arts, in addition to those already described, without departing
20 from the basic inventive concepts. For example, multiple stages of cyanide leaching may be conducted to improve recovery of PGMs and/or base metals. All such variations and modifications are to be considered within the scope of the present invention, the nature of which is to be determined from the foregoing description and the appended claims.

CLAIMS

1. A process for extracting at least one PGM from a source material containing one or more PGMs, the process comprising the steps of:
 - 5 heat-treating the source material to form a residue containing PGMs in a cyanide leachable condition; and
 - cyanide leaching the residue using a solution containing cyanide to form a pregnant cyanide leach liquor containing PGMs in solution.
- 10 2. A process as claimed in claim 1 further comprising the step of repeating the step of cyanide leaching to increase the concentration of PGMs in the cyanide leach liquor.
3. A process as claimed in claim 1 or claim 2 wherein the step of heat-treating is conducted at a temperature in the range of 200 to 550°C.
- 15 4. A process as claimed in claim 3 wherein the step of heat-treating is conducted at a temperature in the range of 275°C to 500°C.
5. A process as claimed in claim 4 wherein the step of heat-treating is conducted at a temperature in the range of 375°C to 425°C.
- 20 6. A process as claimed in claim 4 or claim 5 wherein the step of heat-treating is conducted in an oxidising atmosphere.
7. A process as claimed in claim 1 or claim 2 wherein the step of heat-treating is conducted in a reducing atmosphere at a temperature between 550°C and 1000°C.
- 25 8. A process as claimed in claim 1 or claim 2 wherein the step of heat-treating is conducted in a combination of an oxidising atmosphere and a reducing atmosphere.
9. A process as claimed in any one of the preceding claims wherein the step of cyanide leaching is conducted at a temperature in the range of ambient and 160°C.
- 30

10. A process as claimed in claim 9 wherein the step of cyanide leaching is conducted at a temperature in the range of ambient and 80°C under atmospheric pressure.
11. A process as claimed in claim 9 wherein the step of cyanide leaching is conducted at a temperature in the range of 80°C and 160°C at a pressure of up to 20 bars.
12. A process as claimed in any one of the preceding claims wherein the step of cyanide leaching is conducted for up to 120 hours.
13. A process as claimed in any one of the preceding claims further comprising the step of injecting a source of oxygen during the step of cyanide leaching.
14. A process as claimed in any one of the preceding claims wherein the source material also contains at least one base metal and the process further comprises the step of acid leaching prior to the step of cyanide leaching to form a pregnant acid leach liquor containing at least one base metal in solution.
15. A process as claimed in claim 14 wherein the step of acid leaching is conducted at a temperature between ambient and 200°C and a pressure between atmospheric pressure and 20 bar.
16. A process as claimed in claim 15 wherein the step of acid leaching is conducted at a temperature in the range of ambient and 100°C at atmospheric pressure.
17. A process as claimed in any one of claims 14-16 further comprising the step of recovering base metal from the pregnant acid leach liquor.
18. A process as claimed in any one of the preceding claims wherein the step of cyanide leaching is conducted at a pH within the range of 9 to 12.
19. A process as claimed in any one of the preceding claims further comprising recovering PGMs from the pregnant cyanide leach liquor.

20. A process as claimed in claim 19 wherein PGMs are recovered by reducing the pH of the pregnant cyanide leach liquor to within the range of 1 to 2 to precipitate PGMs.
- 5 21. A process as claimed in any one of the preceding claims wherein the solution containing cyanide contains sodium cyanide and has a cyanide concentration of less than 5% cyanide.
- 10 22. A process as claimed in any one of the preceding claims wherein the solution containing cyanide further comprises lime, caustic soda, peroxide, oxygen, lead nitrate, their derivatives or combinations thereof.
- 15 23. A process as claimed in any one of the preceding claims further comprising the step of crushing and/or grinding the source material prior to the step of heat-treating.
24. A process as claimed in any one of the preceding claims wherein the source material contains gangue and the process further comprises removing at least a portion of the gangue from the source material prior to the step of heat-treating.
- 20 25. A process as claimed in any one of the preceding claims wherein the step of heat-treating is conducted in a fluidised bed or rotary kiln furnace.
- 25 26. A process as claimed in any one of the preceding claims further comprising the step of recovering cyanide from the pregnant cyanide leach liquor for re-use in the process.

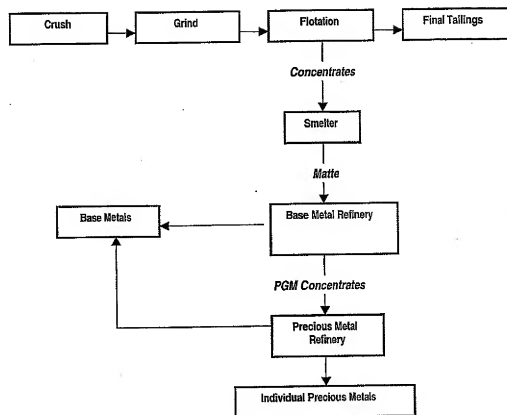
AMENDED CLAIMS

[received by the International Bureau on 23 September 2003 (23.09.03);
original claims 1-20 cancelled; claims 21-26 renumbered as claims 19-24;
remaining claims unchanged]

10. A process as claimed in claim 9 wherein the step of cyanide leaching is conducted at a temperature in the range of ambient and 80°C under atmospheric pressure.
- 5 11. A process as claimed in claim 9 wherein the step of cyanide leaching is conducted at a temperature in the range of 80 °C and 160 °C at a pressure of up to 20 bars.
12. A process as claimed in any one of the preceding claims wherein the step of cyanide leaching is conducted for up to 120 hours.
- 10 13. A process as claimed in any one of the preceding claims further comprising the step of injecting a source of oxygen during the step of cyanide leaching.
14. A process as claimed in any one of the preceding claims wherein the source material also contains at least one base metal and the process further comprises the step of acid leaching prior to the step of cyanide leaching to form a pregnant acid leach liquor containing at least one base metal in solution.
- 15 15. A process as claimed in claim 14 wherein the step of acid leaching is conducted at a temperature between ambient and 200°C and a pressure between atmospheric pressure and 20 bar.
- 20 16. A process as claimed in claim 15 wherein the step of acid leaching is conducted at a temperature in the range of ambient and 100°C at atmospheric pressure.
- 25 17. A process as claimed in any one of claims 14-16 further comprising the step of recovering base metal from the pregnant acid leach liquor.
18. A process as claimed in any one of the preceding claims wherein the step of cyanide leaching is conducted at a pH within the range of 9 to 12.
- 30 19. A process as claimed in any one of the preceding claims wherein the solution containing cyanide contains sodium cyanide and has a cyanide concentration of less than 5% cyanide.

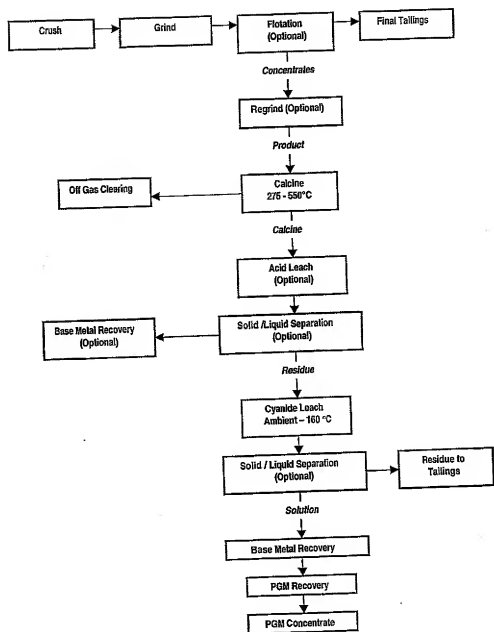
20. A process as claimed in any one of the preceding claims wherein the solution containing cyanide further comprises lime, caustic soda, peroxide, oxygen, lead nitrate, their derivatives or combinations thereof.
- 5 21. A process as claimed in any one of the preceding claims further comprising the step of crushing and/or grinding the source material prior to the step of heat-treating.
22. A process as claimed in any one of the preceding claims wherein the source
10 material contains gangue and the process further comprises removing at least a portion of the gangue from the source material prior to the step of heat-treating.
23. A process as claimed in any one of the preceding claims wherein the step of heat-treating is conducted in a fluidised bed or rotary kiln furnace.
- 15 24. A process as claimed in any one of the preceding claims further comprising the step of recovering cyanide from the pregnant cyanide leach liquor for re-use in the process.

Figure 1



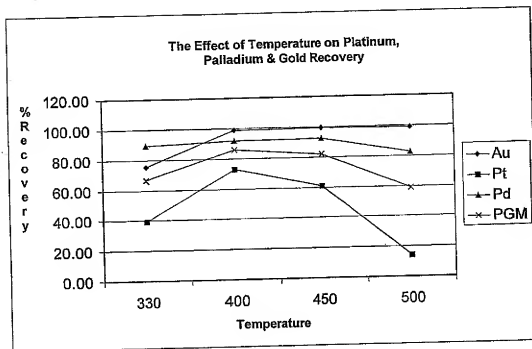
2 / 17

Figure 2



3 / 17

Figure 3

P80 - 53 μm

Calcine - 2 hours various temperatures

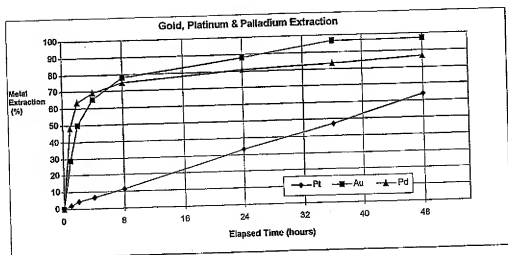
Cyanide leach - 60°C, 48 hours, pH 9.5

P80 - 24 μm

Second cyanide leach

4 / 17

Figure 4

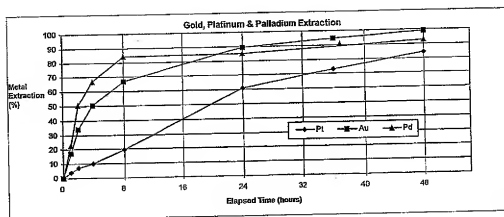
P80 - 53 μm

Calcine - 2 hours at 400°C

No Regrind

5 / 17

Figure 5

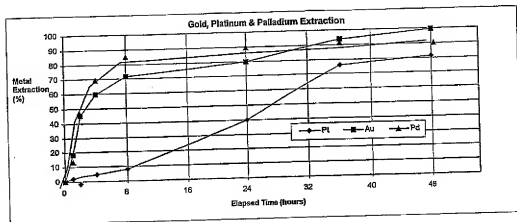
P80 - 53 μ m

Calcine - 2 hours at 400°C

Regrind - 33.5 μ m

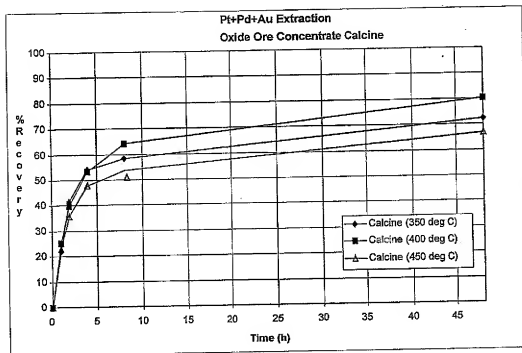
6/17

Figure 6

P80 - 53 μ m

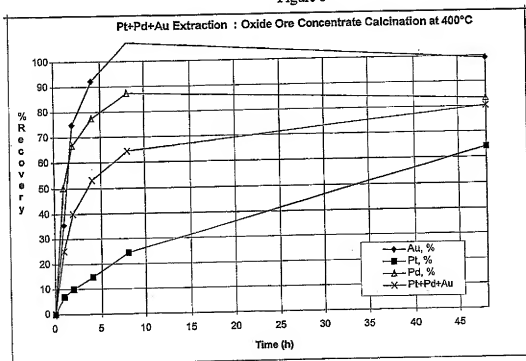
Calcine - 2 hours at 400°C

Regrind - 12.8 μ m

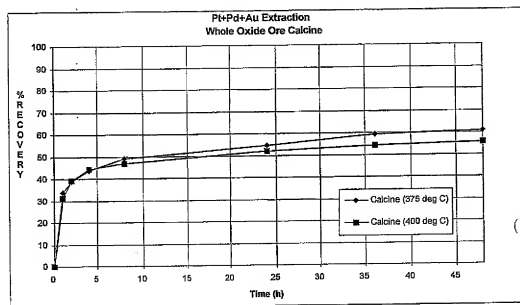
7/17
Figure 7

8 / 17

Figure 8

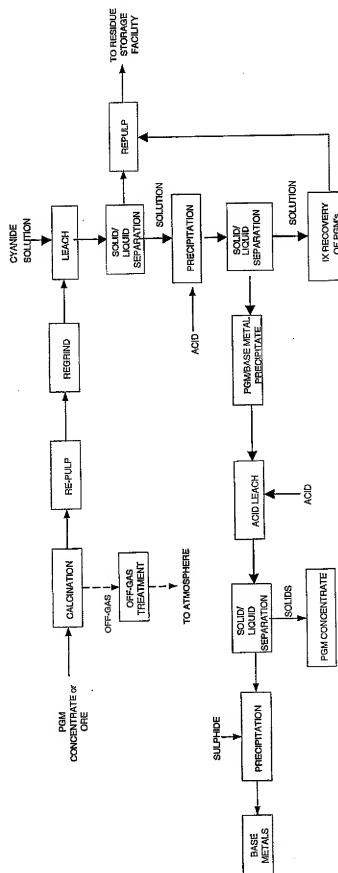


9 / 17
Figure 9



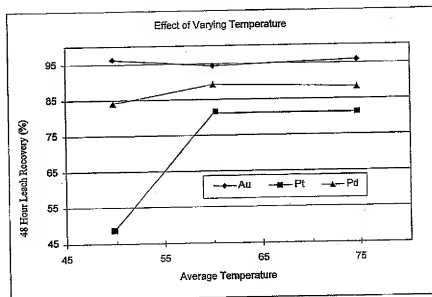
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FIGURE 10



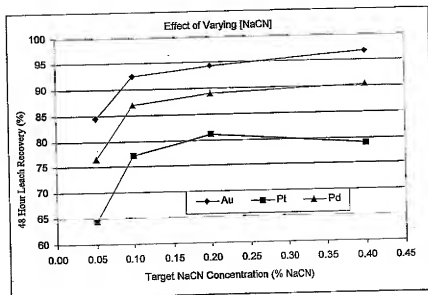
11 / 17

Figure 11



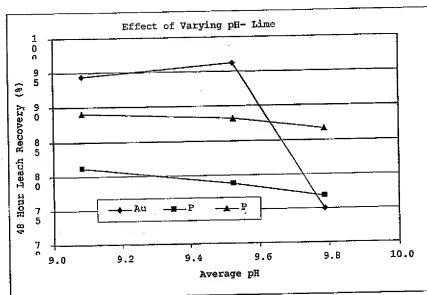
12 / 17

Figure 12



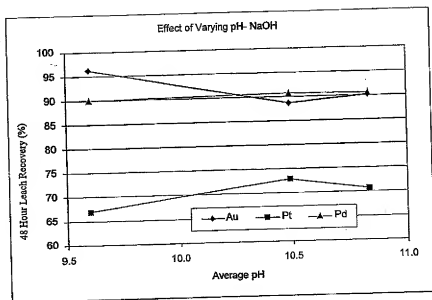
13 / 17

Figure 13



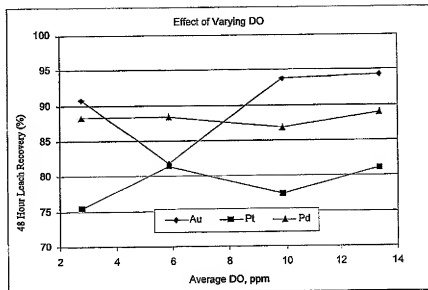
14/17

Figure 14



15 / 17

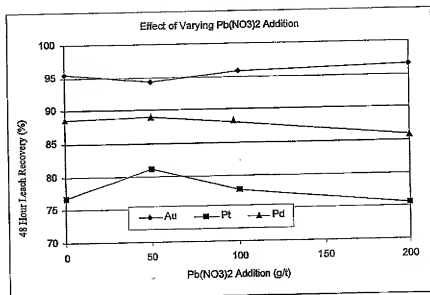
Figure 15



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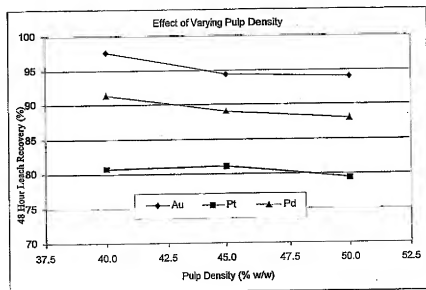
16 / 17

Figure 16



17 / 17

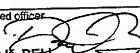
Figure 17



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INTERNATIONAL SEARCH REPORT

International application No.
PCT/AU03/00435

A. CLASSIFICATION OF SUBJECT MATTER		
Int. Cl. ⁷ : C22B 3/12, 3/00, 11/08		
According to International Patent Classification (IPC) or to both national classification and IPC		
B. FIELDS SEARCHED		
Minimum documentation searched (classification system followed by classification symbols) IPC ⁷ As Above		
Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched		
Electronic data base consulted during the international search (name of data base and, where practicable, search terms used) Derwent WPI: IPC ⁷ as above and platinum or PGM+ or Palladium or Rhodium or Ruthenium or Osmium or Iridium		
C. DOCUMENTS CONSIDERED TO BE RELEVANT		
Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	US 3958985 A (Anderson) 25 May 1976 Whole Document	1 to 26
X	CA 2204424 A (De Souza Costa) 3 November 1997 Whole Document	1 to 26
A	GB 2335926 A (Lee Fisher Robinson) 6 October 1999 Whole Document	
<input type="checkbox"/> Further documents are listed in the continuation of Box C <input checked="" type="checkbox"/> See patent family annex		
<p>* Special categories of cited documents:</p> <p>"A" document defining the general state of the art which is not considered to be of particular relevance</p> <p>"E" earlier application or patent but published on or after the international filing date</p> <p>"L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)</p> <p>"O" document referring to an oral disclosure, use, exhibition or other means</p> <p>"P" document published prior to the international filing date but later than the priority date claimed</p> <p>"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention</p> <p>"X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone</p> <p>"Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art</p> <p>"Z" document member of the same patent family</p>		
Date of the actual completion of the international search 7 May 2003		Date of mailing of the international search report 13 MAY 2003
Name and mailing address of the ISA/AU AUSTRALIAN PATENT OFFICE PO BOX 200, WODEN ACT 2606, AUSTRALIA E-mail address: pct@ipaustalia.gov.au Facsimile No. (02) 6285 3929		Authorized officer  DAVID K. BELL Telephone No : (02) 6283 2309

INTERNATIONAL SEARCH REPORT

Information on patent family members

International application No.

PCT/AU03/00435

This Annex lists the known "A" publication level patent family members relating to the patent documents cited in the above-mentioned international search report. The Australian Patent Office is in no way liable for these particulars which are merely given for the purpose of information.

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END OF ANNEX					